इंटरनेट मानक

Disclosure to Promote the Right To Information

Whereas the Parliament of India has set out to provide a practical regime of right to information for citizens to secure access to information under the control of public authorities, in order to promote transparency and accountability in the working of every public authority, and whereas the attached publication of the Bureau of Indian Standards is of particular interest to the public, particularly disadvantaged communities and those engaged in the pursuit of education and knowledge, the attached public safety standard is made available to promote the timely dissemination of this information in an accurate manner to the public.

“जानने का अधिकार, जीने का अधिकार”
Mazdoor Kisan Shakti Sangathan
“The Right to Information, The Right to Live”

“पुराने को छोड नये के तरफ”
Jawaharlal Nehru
“Step Out From the Old to the New”

IS 15082 (2001): N, N-Bishydroxyethyl meta toluidine, technical [PCD 9: Organic Chemicals Alcohols and Allied Products and Dye Intermediates]

“ज्ञान से एक नये भारत का निर्माण”
Satyanarayan Gangaram Pitroda
“Invent a New India Using Knowledge”

Act Number 22 of 2005
The Indian Parliament
RIGHT TO INFORMATION

Made Available By
Public.Resource.Org

“ज्ञान एक ऐसा खजाना है जो कभी चुराया नहीं जा सकता है”
Bhartṛhari—Nītiśatakam
“Knowledge is such a treasure which cannot be stolen”

BLANK PAGE

BRUTUM FULMEN

IGNORANTIA JURIS NON EXCUSAT

PROTECTED BY COPYRIGHT

भारतीय मानक

एन, एन-बिसहाइड्रोक्सीइथायिल मेटा टोल्यूडीन, टेक्नीकल — विशिष्टि

Indian Standard

N, N-BISHYDROXYETHYL META TOLUIDINE, TECHNICAL — SPECIFICATION

ICS 71.080.80

© BIS 2001

BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

December 2001

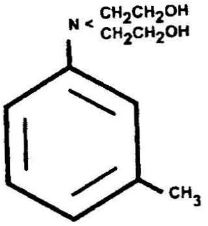
Price Group 3

Dyes Intermediate Sectional Committee, PCD 11

FOREWORD

This Indian Standard was adopted by the Bureau of Indian Standards, after the draft finalized by the Dyes Intermediate Sectional Committee, had been approved by the Petroleum, Coal and Related Products Division Council.

N,N-Bishydroxyethyl meta toluidine ($C_{11}H_{17}NO_2$) is an important intermediate used in the manufacture of azo disperse dyes. It has the following structural formula.



N,N-BISHYDROXYETHYL META TOLUDINE
(Molecular mass 195.0)
(CAS Registry No. [91-99-6]

The composition of the Committee responsible for formulation of this standard is given in Annex B.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

N, N-BISHYDROXYETHYL META TOLUIDINE, TECHNICAL — SPECIFICATION

1 SCOPE

This standard prescribes the requirements and the methods of sampling and test for N,N-Bishydroxyethyl meta toluidine.

2 REFERENCES

The following Indian Standards contain provisions which through reference in this text constitute the provisions of the standards. At the time of publication, the editions indicated were valid. All standards are subject to revisions and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below:

IS No	*Title*
1070 : 1992	Water for general laboratory use (*third revision*)
2552 : 1989	Steel drums (galvanized and ungalvanized) (*third revision*)
5299 : 2001	Methods of sampling and tests for dye intermediates (*first revision*)

3 REQUIREMENTS

3.1 Description

The material shall be in the form of off white solid material.

3.2 The material shall also comply with the requirements given in Table 1.

Table 1 Requirements for N,N-Bishydroxyethyl Meta Toluidine

(*Clauses* 5.3.2 *and* 6.1)

Sl No.	Characteristic	Requirement	Method of Test, Ref to Clause
(1)	(2)	(3)	(4)
i)	Crystallizing temp, °C *Min*	64	9 of IS 5299
ii)	Assay, percent by mass, *Min*	98.0	A-1 & A-3
iii)	N-Hydroxyethyl meta toluidine, *Max*	0.5	A-2 & A-3
iv)	meta-Toluidine	0.3	A-2 & A-3
v)	Moisture, percent by mass, *Max*	1.0	10 of IS 5299
vi)	Insoluble in dil HCl, percent by mass, *Max*	0.3	11 of IS 5299

4 PACKING AND MARKING

4.1 Packing

The material shall be packed in steel drums (*see* IS 2552) lined with suitable polyethylene film or as agreed to between the purchaser and the supplier. Each container shall be securely closed.

4.2 Marking

Each container shall bear legibly and indelibly the following information:

a) Name of the material;
b) Indication of the source of manufacture;
c) Net mass of the material;
d) Batch number; and
e) Month and year of the manufacturer.

4.3 BIS Certification Marking

The container may also be marked with the Standard Mark.

4.3.1 The use of the Standard Mark is governed by the provisions of the *Bureau of Indian Standards Act*, 1986 and Rules and Regulations made thereunder. The details of conditions under which the licence for the use of the Standard Mark may be granted to manufacturers or producers may be obtained from the Bureau of Indian Standards.

5 SAMPLING

5.1 Representative samples of the material shall be drawn as prescribed in 4 of IS 5299.

5.2 Number of Tests

5.2.1 Tests for the determination of N,N-Bishydroxyethyl meta toluidine, N-Hydroxyethyl meta toluidine and meta toluidine shall be conducted on each of the individual samples.

5.2.2 Tests for the determination of remaining characteristics, namely, description, melting range, moisture and solubility shall be conducted on the composite sample.

5.3 Criteria for Conformity

5.3.1 *For Individual Samples*

The lot shall be declared as conforming to the requirement of N,N-Bishydroxyethyl meta toluidine, N-Ethylhydroxyethyl meta toluidine and meta toluidine if each of the individual test results satisfies the relevant requirements given in Table 1.

5.3.2 *For Composite Sample*

For declaring the conformity of the lot to the requirements of all other characteristics (*see* **5.2.2**) tested on the composite sample, the test results for each of the characteristics shall satisfy the relevant requirements given in **3.1** and Table 1.

6 TEST METHODS

6.1 Tests shall be carried out according to the methods refered in col 4 of Table 1.

6.2 Quality of Reagents

Unless specified otherwise, pure chemicals and distilled water (*see* IS 1070) shall be employed in tests.

NOTE —'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

ANNEX A

[*Table* 1, *Sl No.* (ii), (iii) *and* (iv)]

DETERMINATION OF N, N-BISHYDROXYETHYL META TOLUIDINE AND ITS IMPURITIES

A-0 GENERAL

Two methods namely 'Titration Method' (**A-1**) and, Gas Liquid Chromatography (GLC) (**A-3**) have been prescribed for the determination of N,N-Bishydroxyethyl meta toluidine and its impurities. Any of these two methods can be used on routine basis. However in the event of dispute, GLC method shall be treated as referee method.

A-1 TITRATION METHOD

A-1.1 Outline of the Titration Method

A diazonium solution is prepared by reacting 2,4-dinitroaniline in concentrated sulphuric acid with sodium nitrite solution. It is standardized by titrating against N,N-Bishydroxyethyl meta toluidine of known purity. The sample of N,N-Bishydroxyethyl meta toluidine is then titrated against this standard diazonium solution.

A-1.2 Reagents

A-1.2.1 *Glacial Acetic Acid*

A-1.2.2 *Dilute Sulphuric Acid (20 percent m/v)*

A-1.2.3 *Dilute Acetic Acid (20 percent m/v)*

A-1.2.4 *Sodium Acetate Solution (20 percent m/v)*

A-1.2.5 *H-acid Indicator Solution*

Dissolve 0.5 g of H-acid in 100 ml of water containing 1g. of soda ash.

A-1.2.6 *2,4 Dinitroaniline* — 99 percent pure.

A-1.2.7 *N, N-Bishydroxyethyl Meta Toluidine* — reference sample of known purity.

A-1.3 Procedure

A-1.3.1 *Preparation of 2,4 Dinitroaniline Diazo Solution*

Take 600 g (326 ml) of conc H_2SO_4 in a reaction vessel with stirrer which is cooled to 10°C externally on ice bath. Add 69 g of (100 percent) sodium nitrite gradually under stirring maintaining the temperature below 10°C. After the addition is complete, remove the ice bath and bring it to room temperature and then raise to 60°C to dissolve the material completely. After the dissolution is complete the temperature is again brought below 20°C. Similarly, prepare a slurry of 2,4 dinitroaniline in H_2SO_4 by dissolving 183 g of 2, 4 dinitroaniline in 200 g of H_2SO_4 in a 500 ml beaker. Add this slurry to the nitrosulphuric acid slurry prepared earlier maintaining the temperature below 20°C. After the addition is complete, stir for another 2 h for the completion of reaction (stock solution). Depending upon the requirements, a portion of this slurry is taken out and diluted with 10 times by its volume with ice water (for example 10 ml of stock solution and 90 ml of ice water). The solution is then clarified with activated carbon and filtered before use. It is to be noted that the diluted diazo solution is not stable and hence should be prepared fresh every time before use.

A-1.3.2 *Standardization of 2,4 Dinitroaniline Diazonium Solution*

Weigh exactly 0.7809 g. of N,N- Bishydroxyethyl meta toluidine (99 percent) and transfer it to a 1 litre beaker. Dissolve it with 10-15 ml of glacial acetic acid and dilute the solution with ice water to 500 ml. Add about 10 ml. of 20 percent sodium acetate solution (*m/v*) to bring the *p*H between 4 to 6. Titrate this solution with 2,4 dinitroaniline diazo (diluted) until the solution shows the presence of an excess diazo with H-acid (bluish black coloration). This is checked by taking a drop of the reaction mixture and a drop of a diluted solution of H-acid on a piece of filter paper. The bluish black coloration in the junction of the two liquids indicates that the end point is reached. Throughout the titration, the *p*H should be maintained between 4 to 6.

A-1.3.3 Repeat the titration as prescribed in **A-1.3.2** using the test sample of N, N-Bishydroxy meta toluidine.

A-1.4 Calculation

A-1.4.1 Normality of diazonium salt $N = \frac{P \times 0.780}{V \times 19.5}$

where

P = purity (percent by mass) of the known sample, and

V = volume in ml of diazonium solution required for the known sample.

A-1.4.2 Assay, percent by mass = $\frac{V_1 \times N \times 19.5}{0.780}$

where

V_1 = volume in ml of diazonium solution required for the test sample, and

N = normality of diazonium solution.

A-2 DETERMINATION OF N-HYDROXYETHYL META TOLUIDINE AND META TOLUIDIENE BY THIN LAYER CHROMATOGRAPHY

A-2.1 The impurities, namely, N-hydroxyethyl meta toluidine and meta toluidine can be determined by Thin Layer Chromatography as well as by Gas Liquid Chromatography method as given under **A-3**. In case of dispute Gas Liquid Chromatography method shall be considered as referee method. For details of test methods TLC or GC, reference may be made to IS 5299.

A-2.2 Thin Layer Chromatographic Method

A-2.2.1 *Apparatus*

A-2.2.2 *Thin Layer Chromatographic Chamber 25 cm × 25 cm.*

A-2.2.3 *Micropipette* — of 10 microlitre capacity.

A-2.2.4 *Thin Layer Chromatography Plates* — size 20 cm × 20 cm

A-2.2.5 *N-hydroxyethyl Meta Toluidine and Meta Toluidine Pure (Reference Samples)* — Prepare 0.001 percent, 0.002 percent, 0.004 percent solutions in methanol.

A-2.2.6 Eluent ethyl acetate : Toluene (80:20) *v/v*.

A-2.3 Nitrous Fumes Chamber

A glass chamber of size 25 cm × 25 cm with a beaker of capacity 100 ml containing approx 50 ml of conc HCl (30 percent *m/v*). The nitrous fumes are generated by adding a little solid sodium nitrite everytime before inserting the plate.

A-2.4 Spray Reagent — about 1percent solution of para nitro aniline diazo.

A-2.5 Procedure

Weigh 2 g of the sample under test (100 percent) basis and dissolve in 100 ml methanol. Take a plate coated with silica gel G and spot 10 microlitre of each of the test solution and reference solution along the start line at a distance of 3 cm from one another. Saturate the chromatographic chamber with the eluent. Insert the plate in the chamber and allow to run for about 60 min during which time the solvent front travels approximately 10 cm past the start line. Remove the plate from the chamber dry it in air. After drying spray the plate with para nitroaniline diazo solution when the spots (reddish orange) characteristic of meta toluidine and N-Hydroxyethyl-meta toluidine become visible at *Rf* values of 0.90 and 0.70 respectively.

A-2.6 Reporting

Estimate the quantities of the impurities by comparing the colour intensity of the spots of the material under test with those of the solutions of the reference samples.

A-3 DETEREMINATION OF ASSAY AND IMPURITIES BY GLC METHOD

A-3.0 Outline of the Method

The contents of N, N-Bishydroxyethyl meta toluidine and possible impurities, namely meta toluidine and N-hydroxyethyl meta toluidine are determined by Gas Liquid Chromatographic method.

A-3.1 Apparatus

Any gas chromatograph capable of being operated under conditions suitable for resolving the individual constitutents into distinct peaks may be used. A typical chromatogram obtained with Perkin Elmer instrument and the following conditions are shown in Fig. 1.

Column	
Material	Stainless steel
Length	2 000 mm
Internal diameter	2.75 mm
Stationary phase	10 percent OV 101
Solid Support	on chromosorb W/HP mesh 80/100
Carrier Gas	
Nitrogen Flow rate	30 ml/min
Condition	Over temperature isothermal 180°C
Injection temperature	275°C
Detector temperature	275°C
Detector	Flame ionization
Chart speed	10 mm/min up to 3 min, then 5 mm/min

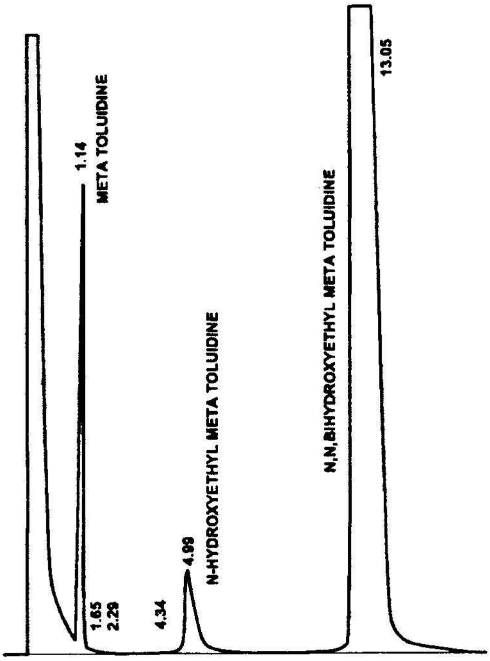


FIG. 1 A TYPICAL CHROMATOGRAM OF N, N-BISHYDROXYETHYL META TOLUIDINE

Sample size	0.4 microlitre of 10 percent solution in acetone.
Retention time	
meta toluidine	1.14 min
N Hydroxyethyl meta toluidine	4.99 min
N, N-Bishydroxyethyl meta-toluidine	13.05 min

A-3.2 Calculation

Follow the given below procedure for calculation when internal standard method is employed.

Percent by mass of N,N-Bishydroxyethyl meta toluidine =

$$\frac{\text{Concentration of standard} \times \text{Area of peak in the sample} \times \text{Strength of standard}}{\text{Concentration of sample} \times \text{Area of peak of standard}}$$

Similarly other fractions can also be calculated.

ANNEX B

(Foreword)

COMMITTEE COMPOSITION

Dyes Intermediate Sectional Committee, PCD 11

Organization	*Representative(s)*
Atul Limited, Valsad, Gujarat	Dr H. Kaiwar (***Chairman***) Shri H. B. Dhuvad (*Alternate* I) Dr J. G. Desai (*Alternate* II) Shri A. R. Desai (*Alternate* III)
Ajanta Chemical Industries, New Delhi	Shri S. D. Bhardwaj Shri Kapil Dev (*Alternate*)
Colour-Chem Ltd, Mumbai	Dr S. Siddhan Shri A. K. Chatterjee (*Alternate* I) Shri K. K. Mehta (*Alternate* II)
Clariant India Ltd, Mumbai	Dr J. N. Shah Shri K. S. Rindani (*Alternate*)
Development Commissioner, Small Scale Industries, New Delhi	Shri S. R. Singh Dr J. S. Rekhi (*Alternate*)
Hindustan Ciba-Geigy India Ltd, Mumbai	Shri S. Rajagopalan Shri D. K. Murthy (*Alternate*)
Hindustan Organic Chemicals Ltd, Rasayani	Shri R. B. Bhandare Dr S. S. Patil (*Alternate*)
Indian Dyestuff Industries Ltd, Mumbai	Dr S. R. Deshmukh Shri S. D. Pawar (*Alternate*)
Indian Chemical Manufacturers Association, Kolkata	Shri G. C. Desai
National Chemical Laboratory, Pune	Dr K. V. Srinivasan
Sudarshan Chemical Industries Ltd, Pune	Shri R. Satyanarayan Shri P. K. Parab (*Alternate*)
The Dyestuff Manufacturer's Association of India, Mumbai	Dr M. G. Narsian Dr S. C. Amin (*Alternate*)
The Gujarat Small Scale Dyestuffs Manufacturer's Association, Ahmedabad	Shri R. S. Patel Shri R. R. Shah (*Alternate*)
BIS Directorate General	Shri Anjan Kar, Director & Head (PCD) [Representing Director General (*Ex-officio*)]

Member-Secretary
Dr (Shrimati) Vijay Malik
Director (PCD), BIS

Bureau of Indian Standards

BIS is a statutory institution established under the *Bureau of Indian Standards Act*, 1986 to promote harmonious development of the activities of standardization, marking and quality certification of goods and attending to connected matters in the country.

Copyright

BIS has the copyright of all its publications. No part of these publications may be reproduced in any form without the prior permission in writing of BIS. This does not preclude the free use, in the course of implementing the standard, of necessary details, such as symbols and sizes, type or grade designations. Enquiries relating to copyright be addressed to the Director (Publications), BIS.

Review of Indian Standards

Amendments are issued to standards as the need arises on the basis of comments. Standards are also reviewed periodically; a standard along with amendments is reaffirmed when such review indicates that no changes are needed; if the review indicates that changes are needed, it is taken up for revision. Users of Indian Standards should ascertain that they are in possession of the latest amendments or edition by referring to the latest issue of 'BIS Catalogue' and 'Standards: Monthly Additions'.

This Indian Standard has been developed from Doc : No. PCD 11 (1304).

Amendments Issued Since Publication

Amend No.	Date of Issue	Text Affected

BUREAU OF INDIAN STANDARDS

Headquarters :

Manak Bhavan, 9 Bahadur Shah Zafar Marg, New Delhi 110 002
Telephones : 323 01 31, 323 33 75, 323 94 02

Telegrams : Manaksanstha
(Common to all offices)

Regional Offices : Telephone

Central : Manak Bhavan, 9 Bahadur Shah Zafar Marg
NEW DELHI 110 002
323 76 17
323 38 41

Eastern : 1/14 C.I.T. Scheme VII M, V. I. P. Road, Kankurgachi
KOLKATA 700 054
337 84 99, 337 85 61
337 86 26, 337 91 20

Northern : SCO 335-336, Sector 34-A, CHANDIGARH 160 022
60 38 43
60 20 25

Southern : C.I.T. Campus, IV Cross Road, CHENNAI 600 113
254 12 16, 254 14 42
254 25 19, 254 13 15

Western : Manakalaya, E9 MIDC, Marol, Andheri (East)
MUMBAI 400 093
832 92 95, 832 78 58
832 78 91, 832 78 92

Branches : AHMEDABAD. BANGALORE. BHOPAL. BHUBANESHWAR. COIMBATORE. FARIDABAD. GHAZIABAD. GUWAHATI. HYDERABAD. JAIPUR. KANPUR. LUCKNOW. NAGPUR. NALAGARH. PATNA. PUNE. RAJKOT. THIRUVANANTHAPURAM.

Printed at Prabhat Offset Press, New Delhi-2